

THE MODEL-BASED CONTROL OF THE GRAIN SIZE OF THE FINAL ANNEALED AUSTENITIC STAINLESS STEELS

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Abstract

The model-based control of the final grain size implemented in the final annealing process of stainless steel is presented. The grain size is an important material property for the formability of stainless steel. The strength and hardness level can also be controlled by the grain size. Therefore, an accurate grain size control is an essential part of a high-quality annealing process.

The grain growth behaviour of most of the austenitic steel grades produced was modelled by numerous isothermal heating tests. The complex time-temperature dependence was successfully formulated. The final calculation with the steel grade dependent parameters was implemented in the modern automation system of the annealing line. The on-line calculation supplies the furnace control with the heating parameters that produce the correct heating cycle for different steel characteristics under various process conditions.

The standard deviation of 0.2 ASTM-units is achieved with the model-based control when processing different steel grades. In addition to the set-point calculation the same model is used for on-line monitoring of the grain size. As a result of model-based monitoring, the inaccurate grain size measurement device that also required lots of maintenance work was removed from the line.

Compared to the table-based set-point calculation, the model-based control is more flexible to react to the customer requirements. Furthermore, the general theoretical model is easy to implement in other annealing lines.

Introduction

This paper presents the model-based control of the final annealing process of stainless steel, based on the physical model of grain size growth. By having a constant grain size, the formability and hardness of the finished material could be guaranteed. In fact, the grain size growth has to be controlled, because many customers tend to order material with a special grain size target.

The grain size control could also be based on the on-line grain size measurement or grade-dependent preset tables. Compared to on-line measurement this solution is probably less expensive for new annealing lines and easier to take into use in old annealing lines. The tuning of preset tables requires more testing than a general model-based solution and is line-specific.

Modelling

When considering isothermal heating and initial grain size after recrystallization, the final grain size after certain annealing time can be calculated by using formulas 1 and 2.¹

$$d_f = (K^{1/n} t_h + d_i^{1/n})^n \text{ where} \quad (1)$$

n = constant

t_h = annealing time

d_i = initial grain size

$$K = A e^{-\frac{Q}{RT}} \text{ where} \quad (2)$$

A = constant

R = gas constant

Q = activation energy

In order to determine the parameters in the formulas 1 and 2, isothermal heating tests with different final temperatures and annealing times were carried out in the thermo-mechanical simulator Gleeble 1500.² The resistive heating up in 1 second fulfilled the requirement of the isothermal condition. The heating trials were repeated for six main grades, covering most of the austenitic product mix at the Tornio plant. The final grain size of the numerous isothermally heated samples were determined by line analysis according to ASTM E-122-88.

In figure 1, the final grain size as a function of annealing time and isothermal temperature is simulated by using the model and parameters that were determined in Gleeble tests. In the model, the initial grain size formed during recrystallization is set to be constant 3.0 μm.³ The different grain size growth between the two grades is significant, emphasizing a need of alloy-dependent control. Furthermore, the hardness as a function of the grain size was modelled in the study. As expected, there was a good correlation between grain size and hardness that could be modelled by exponential function as shown in the figure 1. However, the significant grade effect on the hardness has to be considered when ordering material hardness by the grain size target.

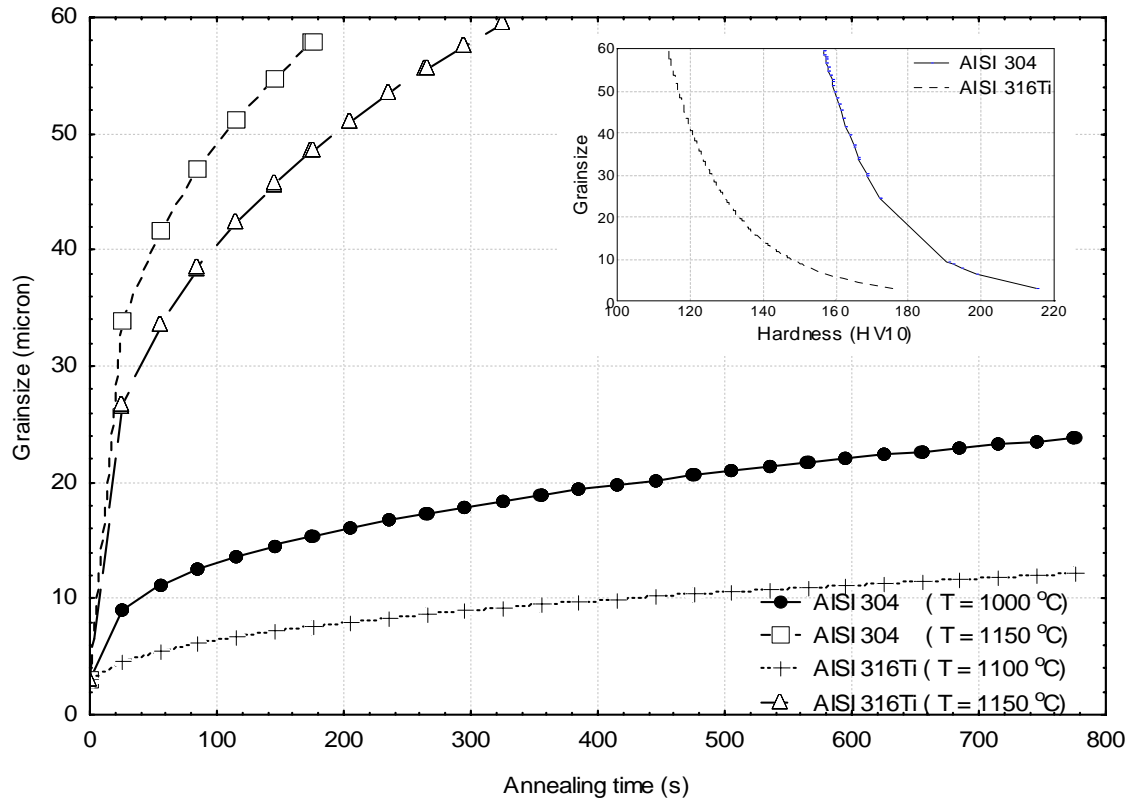


Figure 1. Simulated grain size growth and softening of the steel grades

Implementation of the model in the annealing line

The following aspects were considered during the planning phase of the model-based control.

1. The order-based grain size control
2. Strip temperature model for the furnace atmosphere control
3. Fast and reliable calculation algorithm
4. On-line control of the furnace in order to handle dimension changes
5. Parametrization for the offline adaptation
6. Quality monitoring

The order-based grain size control means, that at the time of order in-take, a special grain size request is saved to the sales system. After that, the ordered grain size target is automatically transmitted through the plant-level system to the model-based control of the furnace automation.

The basic principle of the calculation is shown in figure 2. The standard way to control a furnace is to have setup tables for the final strip temperatures and the controllable strip temperature is measured by pyrometer. Based on the experience from the old annealing lines, the pyrometers were seen as unreliable and sensitive to disturbances such as the low emissivity of a bright cold rolled surface. For that reason, a strip temperature model was included in the calculation, giving also a possibility to control the furnace atmosphere temperatures.

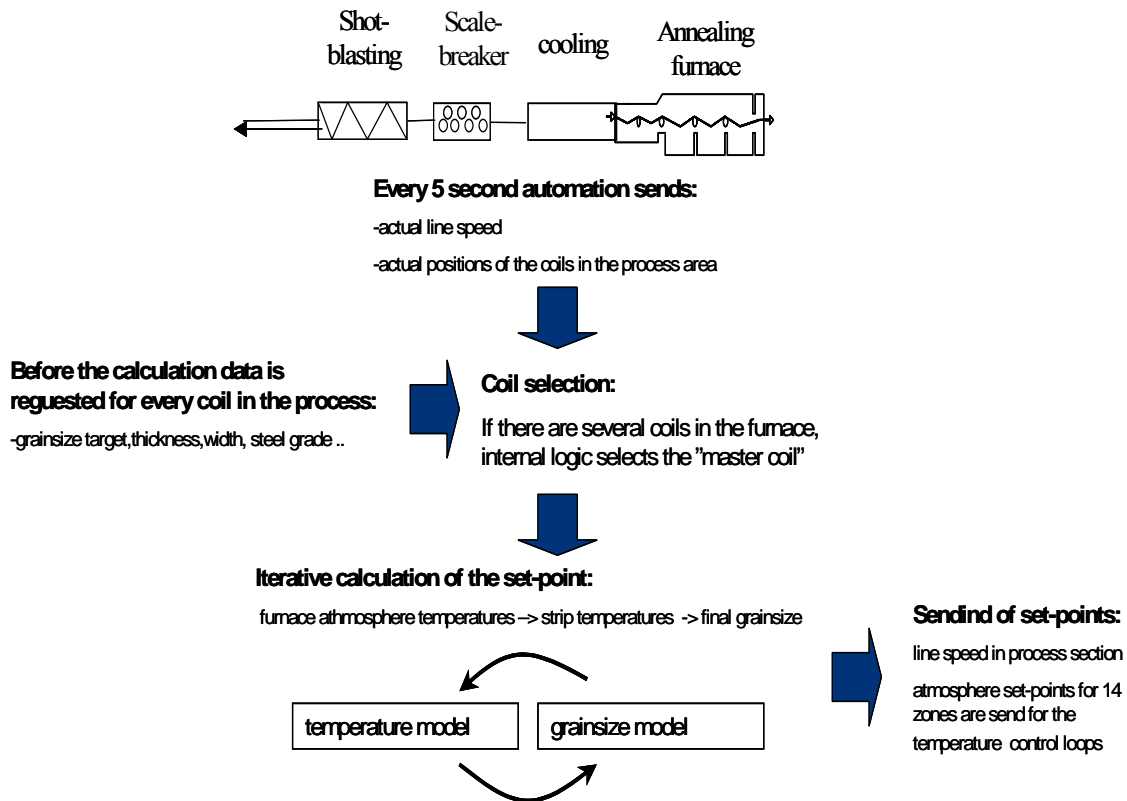


Figure 2. The scheme of model-based control of annealing in the RAP-line

In the long furnaces of the continuous annealing lines, dimensional changes regularly happen. In order to control optimal process speed and furnace temperatures to minimize grain size deviation, the on-line model cyclically calculates new setpoints. To optimize the process, the model has to receive the basic dimensions and order data of all coils in the process area. On-line calculation requires fast calculation routines for the strip temperature and grain size model. That was solved by using Newton's method in the strip temperature calculation and the derivative of a formula of the grain size growth.

The model was highly parametrized so that off-line adaptation could be easily done according to the laboratory analysis of the grain sizes. The possible error could be corrected by changing the emissivity settings of the strip temperature model. Normally the error is related to the drifting behaviour of the temperature measurement in the furnace.

In addition to set-point calculation, the model is used to calculate the final grain size based on the actual line speed and furnace temperatures. For quality monitoring of the calculated grain size, it is allocated to the segments of the strip and sent from automation to the quality reporting system.

Results

The following verification proves that grade-dependent grain size growth can be controlled by the model-based solution. Figure 3 presents the latest grain size analysis for the off-line adaptation. The average error and confidence limits (95 %) are presented in three grade and width groups. The resulted grain size is systematically smaller than the setpoint and the error is correlating to the width of the material. However, the grade effect on the average error is quite

small, especially when considering the error related to grain size analysis. The standard deviation between the grades is about 0.2 ASTM-units.

This example also proves that a regular adaption routine is required when running a process with model-based control. In this case, the width-related problem with the heating power was found and corrective actions were carried out.

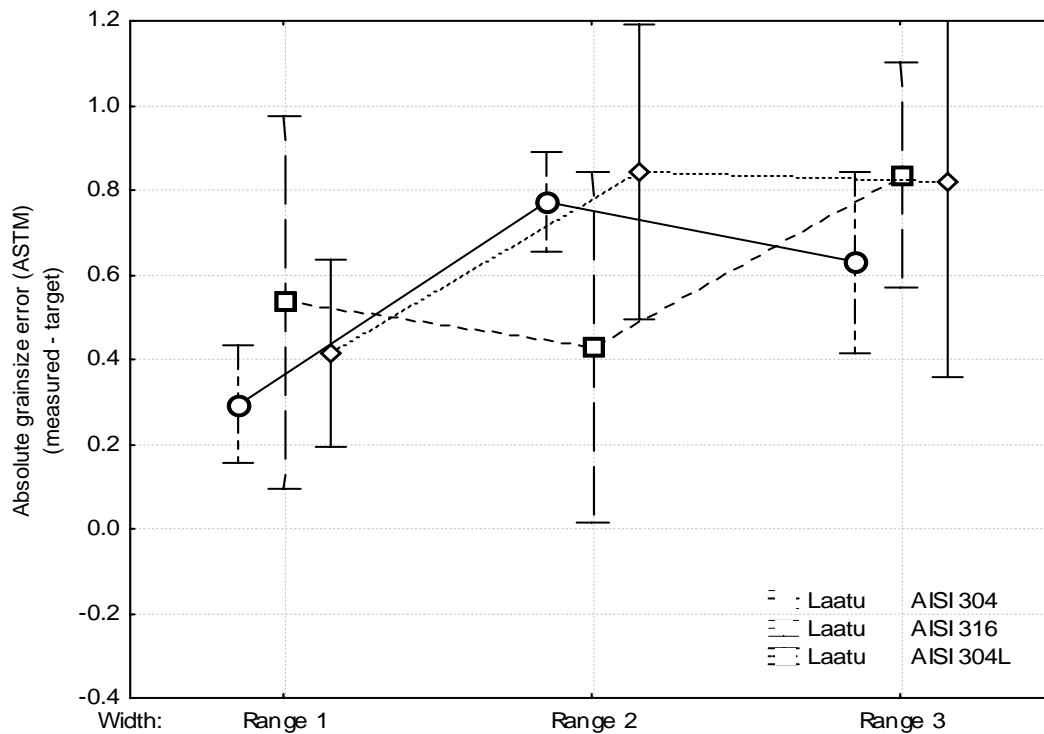


Figure 3. The average and confidence limits (95 %) of the error between measured and target grain size when processing different steel grades at three width ranges (1025, 1275 and 1530 mm)

The first implementation of the grain size model was in the line that was equipped with a grain size measurement device. It was soon discovered, that model results were more accurate and more constant than in-line values received from the ultrasonic-based measurement device. Later on, the device that required lots of maintenance was removed from the line. The good results in the first implementation lead to the decision that the control of the newest annealing line (RAP) in Tornio was only based on the grain size model.

In the RAP-line, acceptable and stable mechanical properties were achieved already in the early stage of the commissioning of the furnace. It was possible to transfer the tested grain size growth model without any modification from the old line to the new annealing line.

Conclusions

- Different stainless steel grades from the 300 series have different rates of grain size growth
- It is possible to model the grain size growth with good accuracy
- The model-based solution that cyclically controls the annealing process by strip speed and furnace atmosphere temperatures, guarantees small deviations of grain size and is flexible to install in new lines

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FABRICATION OF NANOSTRUCTURED STAINLESS STEEL VIA HYDROSTATIC EXTRUSION

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Abstract

In the present study, an 316LVM austenitic stainless steel was processed by hydrostatic extrusion (HE). HE processing results in a microstructure containing nanoscale twins (on average 19 nm in width and 168 nm in length). Annealing was performed after HE at various temperatures for 1 hour. It was found that the annealing at 700°C brings about fully recrystallized microstructure with the mean grain size of 68 nm.

It should be also noted that both types of nanostructures (nanotwins and nanograins) contribute substantially improved mechanical strength. The hydroextrusion increases the microhardness from 348 to 421 HV_{0.2} and an additional annealing at 500°C to 484 HV_{0.2}. At the same time, the ultimate tensile strength becomes improved from 1085 to 1780 MPa after extrusion, decreasing only to 1425 MPa after an additional annealing at 700°C for 1 h. A similar trend is observed for the yield stress. This extremely high strength for the austenitic stainless steel is accompanied by a reasonable level of plasticity (5% elongation in tensile tests).

Introduction

Austenitic stainless steels are widely used in industrial practice. The nanostructural form of these steels is a prospective material for many applications in the nearest future due to the superior properties compared with microcrystalline counterparts. However, the major limitation for a wider use of nano-structured metals is currently the shortage of large-scale, cost-effective methods for their fabrication.

One of the presently investigated techniques for large-scale fabrication of nano-metals is Hydrostatic Extrusion (HE) [1,2]. During HE, the billet, surrounded by pressure transmitted by the liquid medium, is extruded through a die in a frictionless mode and at high strain rates exceeding 10²s⁻¹. Such a high strain rate makes it possible to accumulate a large density of crystal defects at a relatively low total strain, in the range 2-4, making HE one of the promising techniques of Severe Plastic Deformation (SPD). A high strain rate makes the extrusion fast, what is important for industrial applications. The next advantage of HE over other SPD techniques is the possibility to process relatively large-dimension elements of nano-metals, in the form of rods of various cross-sections that can be easily adopted in industrial practice.

Nano-metals produced by HE, as also fabricated by other SPD methods, exhibit extremely high strength but their plasticity is generally far from being satisfactory [3]. In the literature, different ideas have been put forward to retain good plasticity of nano-SPD-metals [4,5,6]. Some authors suggest that a nano-twinned structure is preferable [7]. Others recommend changes in the microstructure by combining SPD with further cold rolling [8]. The aim of this work was to

investigate the nanostructure and properties of 316LVM austenitic stainless steel processed by HE and subjected to post-deformation annealing. The results of tensile tests are discussed in terms of microstructural evolution of nano-316LVM austenitic stainless steel.

Material

The material used in the present study was Sandvic Bioline 316LVM steel of chemical composition shown in Table 1. Samples were hydrostatically extruded in a multi-step process from the initial diameter of 10 mm to the final 4 mm. This corresponds to the true strain of about 2. Cylindrical specimens with the 4 mm height were annealed at different temperatures for one hour. Microstructures were examined using JEOL JEM 1200 EX transmission electron and NIKON EPHIPHOT light microscopes. The revealed microstructures were quantitatively described using a system for image analysis. In the case of nanotwins, width and length were measured, while for regular grains, their equivalent diameter d_2 (defined as the diameter of a circle which has an area equal to the surface area of a given grain) was measured.

Table 1. The chemical composition (wt %) of 316LVM steel

316 LVM	C	Si	Mn	P	S	Cr	Ni	Mo	Cu	N
	0.025	0.6	1.7	0.025	0.003	17.5	13.5	2.8	0.1	< 0.1

The mechanical properties of 316LVM austenitic steel in the microstructural and nanostructural form were determined using an INSTRON 1115 testing machine. Tests were carried out at a strain rate of $8 \times 10^{-4} \text{ s}^{-1}$ using specimens of 2 mm in diameter and 20 mm in the gauge length. In addition, microhardness of the samples was measured using a load of 200 g.

Results

Microstructure observations

The microstructure of HE of 316LVM steel resulted in a significant refinement in HE processing, as shown in Figure 1. Due to low stacking fault energy of this material, nanotwins and shear bands are the most characteristic features of the post-HE microstructure; the twins being on average 19 nm wide and 168 nm in length.

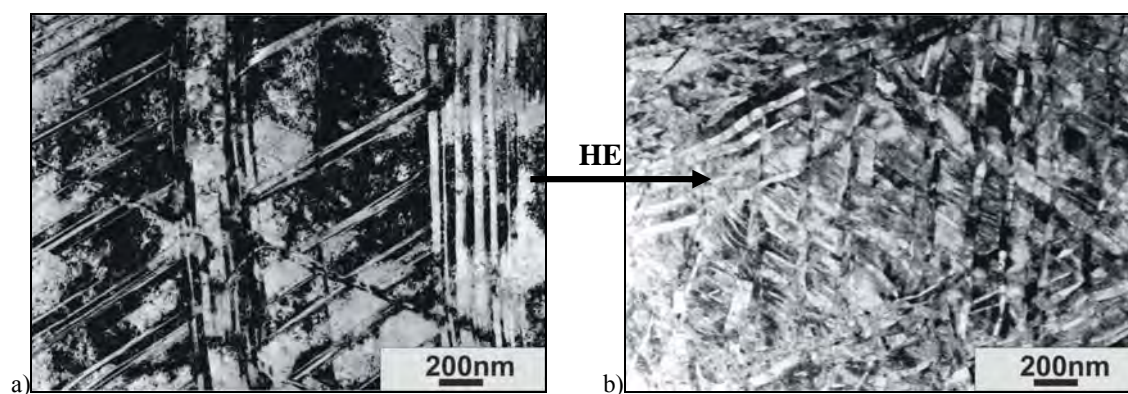


Figure 1. TEM images of the microstructures of austenitic steel 316LVM before (a) and after HE (b).

After annealing at 700°C , a uniform microstructure with fully-recrystallized nanograins was observed (Figure 2a) with the average equivalent grain diameter 68 nm. The grain size distribution for the material recrystallized at 700°C is presented in Figure 3a. It can be noted that nearly all grains have equivalent diameter below 200 nm. Also, one should notice relatively large

spread in the grain size with the coefficient of variation, defined as the ratio of standard deviation to the mean value, $CV = 0.5$.

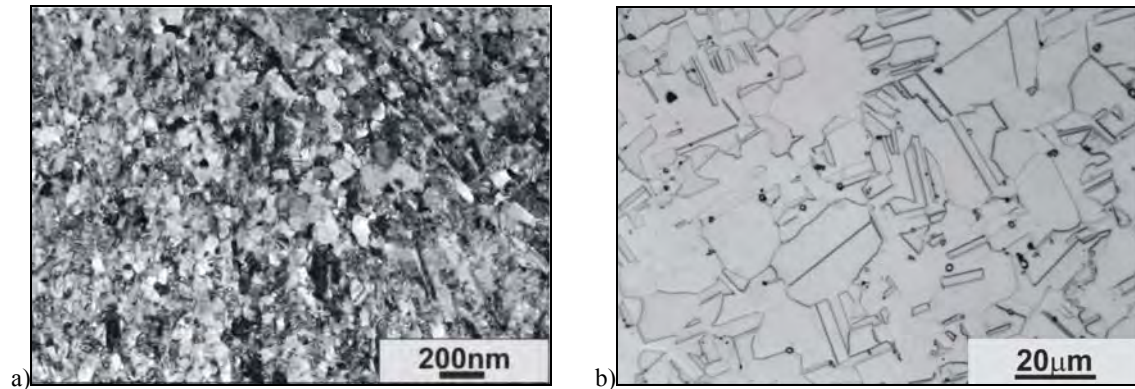


Figure 2. Images of the microstructure of austenitic stainless steel 316LVM after annealing at (a) 700°C and (b) 900°C

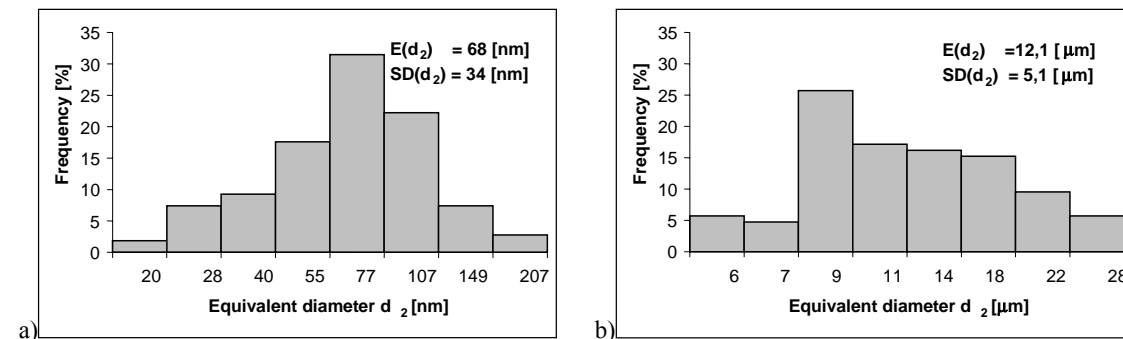


Figure 3. Grain size distribution of austenitic stainless steel 316LVM after annealing at (a) 700°C and (b) 900°C

Observations on the microstructure of the specimens subjected to post-deformation annealing at 800°C indicate that at this temperature some grain growth already takes place leading to the appearance of grains of the size of microns. It has also been found that annealing at 900°C leads to a conventional micro-grained structure with the average equivalent grain diameter 12.1 µm and $CV = 0.42$ (see Figure 2b and 3b). From these results, one can conclude that at the temperatures above 800°C the nanostructure of the 316 LVM stainless steel becomes unstable even for the exposure time of 1 hour.

Microhardness

The microhardness as a function of annealing temperature is shown in Figure 4.

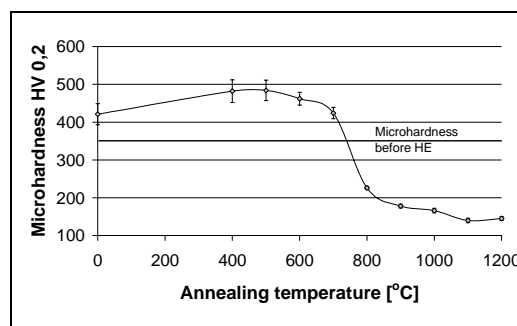


Figure 4. Microhardness as a function of annealing temperature

It can be seen that as a result of HE processing microhardness increases from 348 to 421 HV_{0.2}. Annealing at low temperature results in a slight increase of the microhardness (up to 484 HV_{0.2}). The temperature of fast drop in the microhardness is near 700°C, at which the nano-twinned deformed structure transforms into regular nano-grained structure. The nano-grained structure is still characterized by a high microhardness of 424 HV_{0.2}. A rapid grain growth occurring during annealing at higher temperatures is responsible for a sudden drop in microhardness, for instance to the value of 178 HV_{0.2} after the annealing at 900°C.

Tensile properties

The results of measurements of mechanical properties of 316LVM austenitic stainless steel subjected to different processing steps are shown in Figure 5.

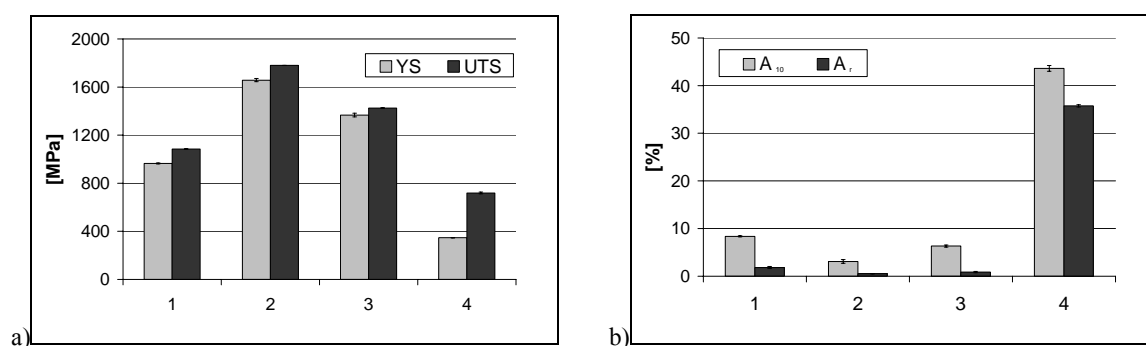


Figure 5. Mechanical properties as a function of processing steps: a) Ultimate Tensile Stress UTS, Yield Stress YS, b) Total Elongation A₁₀, Uniform Elongation A₁; 1 as-received, 2 HE-processed, 3 HE-processed+annealing at 700°C, 4 HE-processed+annealing at 900°C.

It is obvious that HE leads to an increase in mechanical strength. The yield stress rises from 965 to 1658 MPa, whereas the ultimate tensile strength from 1085 to 1780 MPa. At the same time, the uniform elongation A₁₀ drops from 8.4 to 3.1%. Annealing at 700°C after HE results in a slight reductions in the yield and tensile strengths, to 1367 and 1425 MPa, respectively. The plasticity of this nanostructure is twice as high as the plasticity of a nano-twinned one. Annealing at 900°C leads to a drop in strength (UTS = 719 MPa) and increased ductility (the uniform elongation reaches the value of 43.6%).

Summary

In this study, samples 316LVM austenitic stainless steel with two kinds of nanostructure were obtained by HE and by HE followed by annealing. The influence of the type of nanostructure on mechanical properties is clearly noticeable from tensile tests. Although nano-twinned structure reaches high values of the ultimate tensile strength, its plasticity is low. An improvement in plasticity without a considerable lost in the ultimate tensile strength can be achieved by annealing of HE processed samples at 700°C for one hour.

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